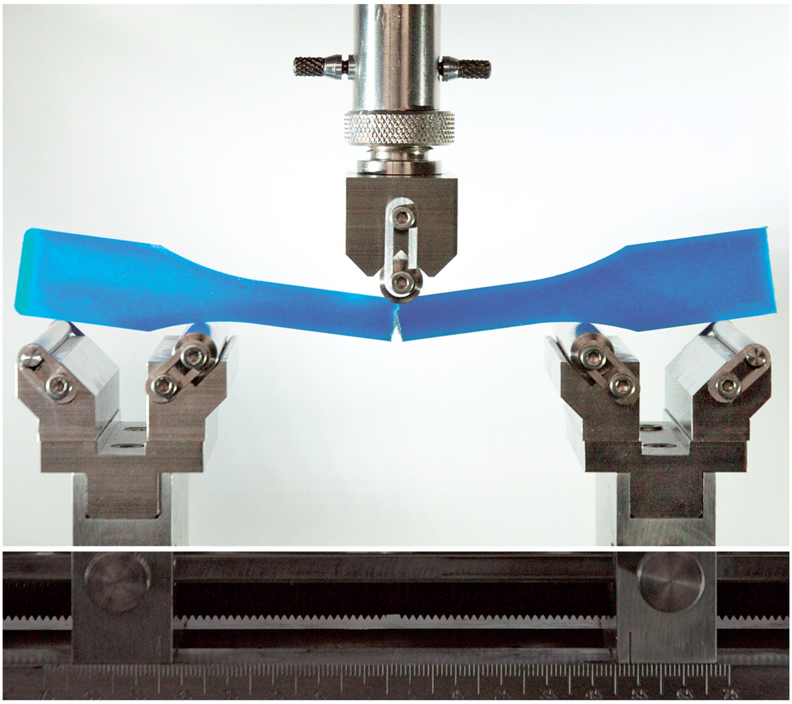
Wolfgang Grellmann
Sabine Seidler

Polymer Testing



2nd Edition

HANSER

Grellmann, Seidler
Polymer Testing

Wolfgang Grellmann
Sabine Seidler (Eds.)

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2nd Edition

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Preface to the Second Edition

The textbook „*Polymer Testing*" is mainly intended for the education of university students and students of universities of applied sciences. This textbook was deemed to be necessary because the testing of polymers has become established as a separate scientific discipline within polymer sciences in recent years. The textbook was first published in German in 2005. An improved English version was published in 2007, and a Russian edition appeared in 2010 with special consideration given to the specific GOST standards.

The positive reviews from our colleagues demonstrate that the concept „Method – Parameters – Examples" meets students` needs and is also accepted in practice.

Although there have been no significant changes to basic testing methods since the first edition appeared, there have been considerable advances in the evaluation of structure-property correlations and standardisation. It has become increasingly necessary to provide material-scientific parameters to quantify the relationship between microstructure and macroscopic properties. Therefore, it seemed necessary to publish a second edition. The previous edition has been comprehensively revised, and the new edition covers all the latest developments in the field, including all amendments to the most important polymer test standards up to May 2013.

Using the same concept and methodical structure in the presentation of polymer test procedures, the parameters obtained by the latter and the selected examples, the new edition provides university students and students of universities of applied sciences with a good and fast source of information. This is why the textbook has been widely adopted by universities and universities of applied sciences for the teaching of „*Polymer Testing*".

In order to provide support the lecturers, a PowerPoint presentation has been created for all pictures and tables. It can be downloaded from www.hanserpublications.com. In this regard, we would like to thank Prof. Dr.-Ing. Christian Bierögel, in particular, for his valuable advice in the preparation of this edition and especially for the new publication of the pictures, which are now in colour, and his extensive work on producing the PowerPoint presentation of all pictures.

A Wiki dictionary, „*Plastics Testing and Diagnostics*“, has been produced on the scientific basis of the book and of publications from the Merseburg scientific school, and it often provides more detail than the book. The dictionary is available at www.polymerservice-merseburg.de/wiki-lexikon-kunststoffpruefung and can be used for practical work. An extensive compilation of fracture mechanics test specimens and approximation equations to calculate parameters in fracture mechanics are just two examples of what the dictionary offers.

We would like to thank Carl Hanser Verlag, especially Ms. Dr. N. Warkotsch, Ms. Dr. C. Strohm, Ms. Dipl.-Ing. (FH) U. Wittmann and Mr. S. Jörg, for their much-appreciated and reliable assistance.

June 2013 The Editors

Preface to the First Edition

This book is based on the editors' extensive experience in research, development and education in the field of materials science and especially polymer testing, polymer diagnostics and failure analysis. The results of their work were published in several reference books about deformation and fracture behavior of polymers, in numerous single publications in peer-reviewed scientific journals and in proceedings. Given the fact that the field of science undergoes a rapid and dynamic development it seemed prudent to present these results in a textbook for students.

The following factors convinced us that a comprehensive representation of the state of knowledge was needed:

- The ever-increasing importance of this materials group for continued technical progress led to an increasing share of polymers and compounds in various applications.
- The increased safety awareness led to the development of hybrid methods of polymer diagnostics, which enable a complex view of the connection between loading and material behavior under actual loading conditions and ambient influences
- As a result of the development of fiber-reinforced thermoplastic and thermosetting composite materials, new challenges to polymer testing methods emerged.
- The increasing use of polymers and elastomers in medical technology for various applications requires the development of technological testing methods for viability, serviceability, operating safety and /or service life.
- As a consequence of the trend to miniaturization components (microsystems), more suitable testing methods are necessary for the evaluation of various thermomechanical loadings of materials properties, e.g., in highly integrated electronic components.

In addition, a number of new standards and regulatory codes for polymer testing have been introduced over the past years, further emphasizing the need for a redesigned textbook for this discipline of science. The book presents a comprehensive representation of knowledge provided by respected colleagues from universities, universities of applied sciences and the polymer industry. A list of co-authors as well as acknowledgements for numerous colleagues and co-workers follow on separate pages.

The editors and co-authors tried hard to overcome the limits of classic polymer testing using ASTM and ISO standards in order to make the importance of polymer testing for the development and application of new polymers, composite materials and materials compounds, as well as the introduction of new technologies, more recognizable.

This book is primarily designed for students of bachelor, diploma and master courses of material science, material technology, plastic technology, mechanical engineering, process engineering and chemical engineering. It can be used by students, teachers of universities and colleges for supplementary studies in the disciplines of chemistry and industrial engineering. The methods of polymer testing are also essential to the development and application of biomedical or nanostructured materials.

With the publication of this book we hope that it will not only serve the important task of training of young scientists in physical and material oriented disciplines, but will also make a contribution to further education of professional polymer testers, design engineers, and technologists.

We thank Carl Hanser Publishers for publishing this book, entitled "Polymer Testing", especially we are grateful to Dr. Christine Strohm who thoroughly revised the complete text for this edition. We also thank Dr. Paul I. Anderson for the translation of several chapters. The main idea of this book was based on the 1992s book by Dr. Heinz Schmiedel "Handbook of Polymer Testing", written in German language. We kept the physical-methodical approach and also, the comprehensive chapter "Fracture Toughness Measurements in Engineering Plastics" based on our research work in this field for many years. For example it is pointed out on the extensive collection of fracture mechanics specimen and the evaluation equations for determination of fracture mechanics parameters.

We want to thank sincerely all co-workers from the Center of Engineering Science and the Institute of Polymer Materials e.V. of the Martin-Luther-University of Halle-Wittenberg and all collaborators from the Institute of Materials Science and Technology of the Vienna University of Technology who, with their commitment and their willing cooperation, made the publication of this book possible in the first place.

Sabine Seidler, Vienna — Wolfgang Grellmann, Halle

May 2007

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The chapters and sections not listed above were written by the editors.

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Mr. Dr. *Hans Walter,* FhG Institute for Reliability and Microintegration (IZM) Berlin, Germany (Chapter 12).

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For the critical revision of single chapters we thank our longtime co-workers Ao. Prof. Dr. mont. *Vasiliki-Maria Archodoulaki*, Dr. *Thomas Koch*, Prof. Dr. *Ines Kotter*, Dr. *Ralf Lach*, Prof. Dr. *Beate Langer* and finally Dr. *Katrin Reincke*.

We thank Ms. *Dagmar Fischer* for the technical editing of figures and images that we provided in various graphical file formats and their transformation into the format required for printing by Carl Hanser Publishers.

Table of Contents

Nomenclature (Selection)

a	(mm)	initial crack length (i.e. machined notch plus razor-sharpened tip), the physical crack size at the start of testing
a_{BS}	(mm)	physical crack length augmented to account for crack tip plastic deformation (fracture mirror length)
a_{cN}	(kJ m^{-2})	*Charpy* impact strength of notched specimen according to ISO 179
a_{cU}	(kJ m^{-2})	*Charpy* impact strength of unnotched specimen according to ISO 179
a_{eff}	(mm)	effective crack length
a/W		ratio of initial crack length to specimen width
$a(\lambda)$		absorption degree
A	(μm)	average interparticle distance
A_0	(mm^2)	cross-section
A_{el}	(N mm)	elastic part of A_G
A_G	(N mm)	total deformation energy of test specimen computed from the area under the load–deflection diagram up to F_{max}
A_H	(N mm)	nominal impact energy of pendulum hammer
A_k	(N mm)	complementary deformation energy, used in the J-integral evaluation method of *Merkle* and *Corten*
A_n		n^{th} amplitude considered for the calculation of the logarithmic decrement
A_{pl}	(N mm)	plastic part of A_G
A_R	(N mm)	crack propagation energy
A_S	(mm^2)	damage area
b	(mm)	specimen width according to ISO 179
b_N	(mm)	remaining width at the notch base of the test specimen according to ISO 179-1
B	(mm)	specimen thickness
C	(mm N^{-1})	compliance
C_i		constants of the power law for describing J_R-curves
d	(mm)	effective way of light through the specimen
D	(μm)	average particle diameter

$D_{1,2}$		geometrical functions in the J-integral evaluation method of *Merkle* and *Corten* (MC)
E	(MPa)	*Young*'s modulus (modulus of elasticity)
E	(kJ m^{-2})	tensile-impact strength according to ISO 8256
E_{50}	(J)	energy at 50 % failure according to ISO 6603-1
E_c	(J)	corrected impact energy according to ISO 179-1
E_d	(MPa)	dynamic flexural modulus
E_f	(MPa)	flexural modulus according to ISO 178
E_n	(kJ m^{-2})	tensile-impact strength (notched specimen) according to ISO 8256
E_t	(MPa)	modulus of elasticity according to ISO 527
F	(mm)	deflection
f_{gy}	(mm)	deflection at the transition from elastic to elastic–plastic material behavior
f_K	(mm)	maximum deflection f_{max} excluding the component f_B
f_{max}	(mm)	deflection at maximum load F_{max}
F	(N)	load (force)
F_1	(N)	inertial load, which arises from the inertia of the part of the test specimen accelerated after the first contact with the striker
F_{gy}	(N)	characteristic load value corresponding to the transition from elastic to elastic–plastic material behavior
F_{max}	(N)	maximum load
F_P	(N)	maximum load (force) according to ISO 6603-2
g		gloss degree
G		gloss
G	(MPa)	shear modulus
G	(N mm^{-1})	energy release rate
G_{12}	(MPa)	interlaminar shear modulus
G_I	(N mm^{-1})	energy release rate in mode I
G_{Ic}	(N mm^{-1})	energy release rate, critical value at the point of unstable crack growth; static loading, geometry-independent
G_{IIc}	(N mm^{-1})	energy release rate in mode II, critical value at the point of unstable crack growth; static loading, geometry-independent
G'	(MPa)	dynamic modulus (storage modulus)
G''	(MPa)	dynamic modulus (loss modulus)
GD		basic dispersion
h		gloss height
H		heterogeneity

HB	(N mm^{-2})	ball indentation hardness according to DIN EN ISO 2039-1
HDT	(°C)	heat distortion temperature according to ISO 75
HK	(N mm^{-2})	*Knoop* hardness
HM	(N mm^{-2})	*Martens* hardness
HR	(N mm^{-2})	*Rockwell* hardness
HV	(N mm^{-2})	*Vickers* hardness
I		intensity
I_p	(A)	photometer current intensity at a bearing specimen
I_{po}	(A)	photometer current intensity at a bearing specimen at perpendicular light direction
I_{sw}	(A)	photometer current intensity at a bearing matt white standard
I_{swo}	(A)	photometer current intensity at a bearing matt white standard at perpendicular light direction
J	(N mm^{-1})	J-integral; a mathematical expression, a line or surface integral that encloses the crack front from one surface to the other, used to characterize the local stress–strain field around the crack front; fracture mechanics parameters are calculated using methods of evaluation of this integral
J_I	(N mm^{-1})	J value in mode I (the index I is only used in the case of geometry independence)
J_{Id}	(N mm^{-1})	critical J value at the point of unstable crack growth; dynamic loading, geometry-independent
J_{Id}^{MC}	(N mm^{-1})	critical J value at the point of unstable crack growth, for dynamic loading, in the geometry-independent J-integral evaluation method of *Merkle* and *Corten*
J_{Id}^{ST}	(N mm^{-1})	critical J value at the point of unstable crack growth, for dynamic loading, in the geometry-independent J-integral evaluation method of *Sumpter* and *Turner*
$J_{0,2}$	(N mm^{-1})	technical crack initiation value for an amount of crack growth of $\Delta a = 0.2$ mm
J_i	(N mm^{-1})	physical crack initiation value determined from intersection of stretch zone width and J–R curve
JT_J	(N mm^{-1})	energy absorption capacity of a material during stable crack growth
k		*Boltzmann* number ($k = 1{,}38 \cdot 10^{-23}$ J K^{-1})
k		number of colour order of an isochromatic line series
K	(MPa)	compression modulus
K	(MPa $mm^{1/2}$)	stress intensity factor
K_I	(MPa $mm^{1/2}$)	stress intensity factor in mode I (the index I is only used in the case of geometry independence)

K_{Ic}	(MPa mm$^{1/2}$)	fracture toughness, critical value at the point of unstable crack growth; static loading, geometry-independent
K_{Id}	(MPa mm$^{1/2}$)	fracture toughness, critical value at the point of unstable crack growth; dynamic loading, geometry-independent
$K_{Ic;Id}^{CTOD}$	(MPa mm$^{1/2}$)	K_{Ic} and K_{Id}, calculated from CTOD
l	(mm)	specimen length
L	(mm)	clamping length; initial distance between grips
L	(mm)	support span according to ISO 179-1
L_0	(mm)	initial gauge length
m	(g)	mass
m		constraint factor in relation between J and δ concepts
m_P	(kg)	weight of pendulum hammer
M_c		molecular weight of a chain network
M_W	(g mol^{-1})	molecular weight, weight average
MFR	(g (10 min)$^{-1}$)	melt mass-flow rate according to ISO 1133
MVR	(cm^3 (10 min)$^{-1}$)	melt volume-flow rate according to ISO 1133
n		rotational factor
n		refraction, refraction index
n_C		refraction at wavelength C (656 nm) of the *Fraunhofer* line
n_D		refraction at wavelength D (589 nm) of the *Fraunhofer* line
n_f		refraction of immersion oil at temperature in contrast minimum
n_F		refraction at wavelength F (486 nm) of the *Fraunhofer* line
n_x		refraction of immersion oil at room temperature
N		crosslink density
p (λ)		spectral reflexion degree
p	(MPa)	pressure
Q	(J)	quantity of heat
r_N	(µm)	notch base radius according to ISO 179-1
R		universal gas constant (R = 8,314 J mol^{-1} K^{-1})
R_s		reflectance of a layer above a black ground
R_∞		reflectance of an optical dense layer
s	(mm)	support span
S		dispersion coefficient
t	(s)	time
t_b	(ms)	time to brittle fracture
t_B	(ms)	time to fracture

t_p	(ms)	time to maximum load according to ISO 6603-2
tan δ		mechanical loss factor
T		total transmission
T	(°C)	temperature
T_D		translucency
T_g	(°C)	glass transition temperature
T_g		haze dimension
T_J		tearing modulus
$T_J^{0,2}$		tearing modulus determined from J–Δa curve at Δa = 0.2 mm
T_m	(°C)	melting temperature
T_p		transparency
T_s		transmittance of the scattered light
T_S	($N\ mm^{-1}$)	tear strength
$T_\delta^{0,2}$		tearing modulus determined from δ-Δa curve at Δa = 0,2 mm
U	(N mm)	deformation energy
v	(mm)	crack-mouth-opening displacement
v_I	($m\ s^{-1}$)	impact velocity according to ISO 13802
v_L	(mm)	load-line displacement
v_T	($mm\ min^{-1}$); ($m\ s^{-1}$)	cross-head speed
V	mm^3	volume
VST	(°C)	*Vicat* softening temperature
W	(mm)	specimen width
W_S	($mm^3\ (Nm)^{-1}$)	specific wear rate
x		standardized colour data
X		intensity of the colour red
y		standardized colour data
Y		intensity of the colour green
z	(mm)	distance of knife-edge from specimen surface
Z		intensity of the colour blue
α	(K^{-1})	linear thermal expansion
β		proportionality constant of geometrical size criterion for LEFM
β	($n\ °C^{-1}$)	temperature coefficient of refraction
γ		shear strain
γ_{12}		interlaminar shear strain
$\dot{\gamma}$		shear rate

δ	(mm)	crack-tip-opening displacement describing the local strain field in front of the crack tip, calculated with the help of the plastic-hinge model
δ_I	(mm)	crack-tip-opening displacement in mode I (the index I is only used in the case of geometry independence)
δ_{Ic}	(mm)	critical δ value for unstable crack growth, quasi-static loading, geometry-independent
δ_{Id}	(mm)	critical δ value for unstable crack growth, dynamic loading, geometry-independent
δ_{Idk}	(mm)	critical δ value for unstable crack growth obtained by using advanced plastic-hinge model, dynamic loading, geometry-independent
$\delta_{0,2}$	(mm)	technical crack-opening displacement calculated at $\Delta a = 0.2$ mm
δ_i	(mm)	crack-tip-opening displacement at physical crack initiation
Δa	(mm)	amount of stable crack growth, distance between original crack size and crack front after loading
Δa_{max}	(mm)	upper validity limit of Δa
Δa_{min}	(mm)	lower validity limit of Δa
Δl	(mm)	increase in specimen length
ΔL	(mm)	increase in clamping length
ΔL_0	(mm)	increase in gauge length
Δn		birefringence
Δt	(s)	time difference
Δv	(m s^{-1})	velocity change
ε		proportionality constant of geometrical size criterion for J-integral concept
ε	(%)	strain
ε	(°)	angle of incidence
ε'	(°)	angle of refraction
$\dot{\varepsilon}$	(s^{-1})	strain rate
ε_{AE}	(%)	critical strain at acoustic onset
ε_B	(%)	tensile strain at break according to ISO 527
ε_f	(%)	normal flexural strain
ε_l	(%)	local strain
ε_{lmax}	(%)	maximum local strain
ε_{lmin}	(%)	minimum local strain
ε_M	(%)	normative strain at tensile strength according to ISO 527
ε_q	(%)	lateral (transverse) strain

ε_t	(%)	nominal tensile strain according to ISO 527
ε_{tB}	(%)	nominal tensile strain at break according to ISO 527
ε_{tM}	(%)	nominal strain at tensile strength according to ISO 527
ε_W	(%)	true strain
ε_y	(%)	yield strain according to ISO 527
η		geometrical function
η		dynamic viscosity
$\eta_{el; pl}$		geometrical functions for assessment of elastic (el) and plastic (pl) parts of deformation energy used in the J-integral evaluation method of *Sumpter* and *Turner*
λ		extension ratio
λ	(W (m K)$^{-1}$)	heat conductivity
λ	(nm)	light wavelength
Λ		logarithmic decrement according to ISO 6721-1
μ		coefficient of friction, *Poisson*'s ratio
ν		*Poisson*'s ratio
ν		*Abbe* number
ξ		proportionality constant of geometrical size criterion for CTOD
ρ	(kg m^{-3})	density
σ	(MPa)	stress
σ_B	(MPa)	tensile stress at break according to ISO 527
σ_f	(MPa)	flexural stress according to ISO 178
σ_{fc}	(MPa)	flexural strength at peripheral strain of 3.5 % according to ISO 178
σ_{fM}	(MPa)	flexural strength according to ISO 178
σ_F	(MPa)	yield stress: either σ_y or $\sigma_F = 1/2(\sigma_y+\sigma_M)$
σ_l	(MPa)	local stress
σ_M	(MPa)	tensile strength according to ISO 527
σ_V	(MPa)	comparative stress
σ_W	(MPa)	true stress
σ_y	(MPa)	yield stress (yield point) according to ISO 527
τ	(MPa)	shear stress
τ		oscillation period
τ_{12}	(MPa)	interlaminar shear stress
τ (λ)		spectral transmittance
φ_V		filler or fiber content
Φ		light beam that is bearing on a layer

Φ_{ds}^{KW}		small angle light scattering
Φ_{ds}^{WW}		wide angle light scattering
Φ_{dp}		linear transmitted part of light
$\Phi_{e}\lambda$	(W)	hitting spectral radiant flux
$(\Phi_{e}\lambda)_{a}$	(W)	absorbed spectral radiant flux
$(\Phi_{e}\lambda)_{p}$	(W)	reflected spectral radiant flux
$(\Phi_{e}\lambda)_{\tau}$	(W)	transmitted spectral radiant flux

Terminology

AE	acoustic emission analysis
AF	aramid-fiber
AFM	atomic force microscopy
ASTM	American Society for Testing and Materials
ATR	attenuated total reflection
BMI	Bismaleinimide
BSS	Boeing Specification Support Standard
BTT	brittle-to-tough transition temperature
CA	coupling agent
CF	carbon-fiber
CFC	carbon-fiber composite
CFRP	carbon-fiber reinforced polymer
CFR	Code of Federal Regulations
CT	compact tension specimen
CTOD	crack-tip-opening displacement
DCB	double-cantilever beam specimen
DENT	double-edge-notched tension specimen
DIN	German Institute of Industrial Standards (Deutsches Institut für Normung)
DMTA	dynamic–mechanical–thermal analysis
DOP	Dioctylphthalat
DSC	differential scanning calorimetry
DTG	differential thermogravimetry
DVM	German Association for Materials Testing (Deutscher Verband für Materialprüfung)
DVS	German Association for Welding (Deutscher Verband für Schweissen und verwandte Verfahren)
EN	European Norm
EPFM	elastic–plastic fracture mechanics
ESIS	European Structural Integrity Society
ESPI	electronic speckle-pattern interferometry
FAR	Federal Aviation Regulations
FEM	finite element method